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Indian Standard

METHOD FOR EVALUATING STRENGTH AND SHADE OF ACID DYES BY DYEING TEST

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METHOD FOR EVALUATING STRENGTH AND SHADE OF ACID DYES BY DYEING TEST

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Indian Standard

METHOD FOR EVALUATING STRENGTH AND SHADE OF ACID DYES BY DYEING TEST

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 11 November 1975, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

0.2 Acid dyes are marketed in large varieties, different strengths and also as mixtures. The method laid down in this standard for determining the strength of dyestuffs against a mutually accepted standard would be useful for assessing both the strength and the shade of the dyestuff on wool.

0.3 The method prescribed in this standard is a general method and wherever special instructions are given by the manufacturers, these should be followed while carrying out the dyeings of the dyestuffs. In order to compare the exhaust property of the dyestuff under test and the standard dyestuff, it is recommended to carry out the exhaust dyeings. It is generally better to use medium shades (about 1 percent) as they show any variations in strength more clearly than dark shades. Wherever necessary, it is advisable to carry out evaluation in deep shades also as recommended by the manufacturers (for example blacks, navy blue, etc).

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes a method for evaluating strength and shade of acid dyes (level-dyeing acid dyes, acid-milling dyes, neutral-dyeing acid dyes, 1 : 1 and 2 : 1 metal complex acid dyes) by dyeing test.

2. SAMPLING

2.1 Lot — All the containers of the same dye and of the same concentration delivered to a buyer against a despatch note shall constitute a *lot*.

*Rules for rounding off numerical values (revised).

2.2 Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from a lot shall be as given below:

<i>Lot Size</i>	<i>Sample Size</i>
Up to 8	2
9 to 15	3
16 to 25	4
25 and above	5

2.3 From each container draw small quantities of dye by a suitable sampling instrument from at least 3 different parts and mix them thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

3. STANDARD DYESTUFF

3.1 The standard sample of dyestuff against which the strength of dyestuffs under test is evaluated shall be as agreed to between the buyer and the seller.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5. EVALUATION OF STRENGTH OF DYESTUFF

5.1 Preliminary Test to Determine Strength— Before dyeing the hanks, carry out a preliminary test to determine the approximate strength of the dyestuff under test as given in 5.1.1.

5.1.1 Take separately 10 ml of 0.1 percent solutions of the dyestuff under test and the standard dyestuff (*see* A-4.1). Put a spot or streak or pour each of the solutions of the dyestuff under test and the standard dyestuff on filter paper. Visually, examine the spots. If they are not equal in strength, dilute the stronger solution with water to such an extent as to get approximate equal strength of dyestuff spotted on filter paper. Calculate the approximate strength of the dyestuff under test by the following formula:

$$\text{Approximate strength (} S_a \text{) of dyestuff under test} = \frac{V_t \times 100}{V_s}$$

where

V_t = final volume in millilitres of the solution of dyestuff under test, and

V_s = final volume in millilitres of the solution of the standard dyestuff.

*Specification for water, distilled quality (*revised*).

5.2 Prepare solutions of standard dyestuff and dyestuff under test as given in **A-4.1**.

5.3 Prepare dyeings of 0.95, 1.00 and 1.05 percent depths of shade of the standard dyestuff as given in **A-4.2.1**.

5.4 Similarly, prepare dyeings of 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade of dyestuff under test as given in **A-4.2.2**.

5.5 Carry out the dyeings of the fresh hanks in the exhaust liquors of the above dyeings by following the procedure given in **A-4.3**.

5.6 Assessment — Compare the dyeings of dyestuff under test (*see* **5.4**) and standard dyestuff (*see* **5.3**) in north skylight (*see* Note). Select a dyeing of the dyestuff under test which exactly matches with one of the dyeings of the standard dyestuff. Note the percentages of the dyeings which match exactly.

NOTE — Before comparing the dyeings, they should be spread out properly. The dried hanks should be laid side by side in the same plane and oriented in the same direction. They should be combed to a uniform thickness to avoid the effects of the backing on the appearance. The consistency in strength variation of different dyeings of standard dyestuff and the dyestuff under test should be observed. If the strength variation between the two consecutive dyeings are not constant, the dyeings should be repeated.

5.7 For Deep Shades — Wherever necessary, evaluation shall also be carried out in deep shades on similar lines as for one percent shade, using dyeing assistants in accordance with the manufacturers' recommendations.

6. CALCULATIONS

6.1 Calculate the strength of the dyestuff under test by the following formula:

$$S = A/B \times S_a$$

where

S = strength of dyestuff in percentage,

A = percentage shade of the standard dyestuff,

B = percentage shade of the dyestuff under test matching with A , and

S_a = approximate strength of the dyestuff under test as determined under **5.1.1**.

7. REPORT

7.1 The report shall include the following information:

- The strength of dyestuff in percent (*see* **6.1**),
- Shade of the dyestuff in comparison with that of the standard dyestuff, and
- Shade of the exhaust dyeings in comparison with that of the exhaust dyeing of the standard dyestuff.

APPENDIX A

(*Clauses 5.1.1, 5.2, 5.3, 5.4 and 5.5*)

GENERAL METHOD FOR DYEING OF ACID DYES ON WOOL

A-1. APPARATUS

A-1.1 Dye Vessel — porcelain or stainless steel dye vessels.

A-1.2 Graduated Pipettes — capable of measuring correct up to 0.1 ml.

A-2. DYEING ASSISTANTS

A-2.1 Water — Distilled water (*see* IS : 1070-1960*) shall be used in the preparation of the dye-bath.

NOTE — For rinsing, water having hardness of not more than 50 ppm expressed as calcium carbonate may be used.

A-2.2 Sodium Sulphate (Calcined) Solution — 10 percent (*m/v*) (*see* IS : 255-1967†).

NOTE — Quality of sodium sulphate is extremely important. Commercial brands may be acidic or alkaline, therefore, they shall be neutralized.

A-2.3 Acetic Acid Solution — 10 percent (*m/v*) (*see* IS : 695-1967‡).

A-2.4 Formic Acid Solution — 10 percent (*m/v*).

A-2.5 Sulphuric Acid Solution — 10 percent (*m/v*) (*see* IS : 266-1961§).

A-2.6 Ammonium Sulphate — 10 percent (*m/v*).

A-2.7 Ammonium Acetate — 10 percent (*m/v*).

A-3. PREPARATION OF HANKS FOR DYEING

A-3.1 A sufficient number of hanks of scoured, 100 percent pure wool knitting yarn having no finishing chemical, blueing agent or fluorescent brightening agent shall be used. Each hank shall weigh 5.0 ± 0.1 g (*see* Note).

NOTE — If the mass of the hank is not 5.0 ± 0.1 g, then it shall be weighed accurately and the amount of dyestuff and the chemicals to be taken shall be calculated accordingly.

*Specification for water, distilled quality (*revised*).

†Specification for sodium sulphate, anhydrous (*first revision*).

‡Specification for acetic acid (*first revision*).

§Specification for sulphuric acid (*revised*).

A-3.2 Preparation of Test Hanks — Treat the required number of hanks in warm water (not higher than 60°C) for 10 minutes, squeeze evenly to contain approximately its own mass of water, cool and enter into the dye-bath.

A-4. PROCEDURE

A-4.1 Preparation of Dyestuff Solutions

A-4.1.1 Weigh accurately 1.00 g of standard dyestuff. Paste it thoroughly with cold water. Add hot water and dissolve the dyestuff. If necessary, heat the solution till it becomes clear. Dilute the solution to 1 000 ml with cold water.

A-4.1.2 Similarly weigh accurately $\frac{1.00 \times 100}{S_u}$ g of dyestuff under test (*see* 5.1.1) and prepare solution following by the procedure given in A-4.1.1.

A-4.2 Dyeing (for 5.0 ± 0.1 g Hank)

A-4.2.1 Pipette out separately 47.5, 50.0 and 52.5 ml of standard dyestuff solution in the dye vessels so as to give 0.95, 1.00 and 1.05 percent depths of shade. Add requisite quantity of water deducting the amount of dyeing assistant solutions to make the final volume to 300 ml (liquor to material ratio 60:1). Add requisite quantity of sodium sulphate (calcined) solution wherever necessary and half the quantites of acid solution or acid salt solution depending on the nature of the dyestuff (*see* Note 1). Stir the dye liquor and raise the temperature to 50°C. Enter the wetted hanks and turn them frequently so as to obtain level dyeings. Work for 10 minutes at this temperature. Remove the hank from the dye-bath, add second lot of requisite quantity of acid solution or acid salt solution and stir well. Enter the hanks into the dye-bath and turn the hanks frequently. Slowly raise the temperature of water-bath to boil within 20 minutes. Continue the dyeing at boil for 45 to 60 minutes (*see* Note 2). Remove the hank. If necessary, add weak or strong acid solution (*see* Note 1). After addition, stir the bath and enter the hanks. Continue the dyeing further for 15 minutes. At the end of dyeing squeeze the dyed hank returning the squeezed out solutions to the respective dye-baths. Then rinse the dyed yarn with warm water (50 to 60°C). Dry the hanks in oven or in drier at a temperature not exceeding 70°C.

NOTE 1 — Addition of strong acid, weak acid or acid salt individually or in combination should be made with or without sodium sulphate (calcined) according to the recommendations given below or according to the recommendations of the dyestuff manufacturers or by the mutual agreement between the buyers and the sellers. Recommended quantities of dyeing assistants for medium shades (about 1 percent shade) are given below:

- a) *Level dyeing acid dyes* — 5 ml sodium sulphate (calcined) solution and 1.75 ml sulphuric acid solution or formic acid solution.

b) *Acid milling dyes and neutral dyeing dyes*

- 1) 5 ml sodium sulphate (calcined) solution and 0.75 ml acetic acid solution.

If necessary, for exhaustion, add additional amount of 0.75 ml sulphuric acid solution or formic acid solution. Continue the dyeing further according to the recommended procedure.

OR

- 2) 5 ml sodium sulphate (calcined) solution and 3 ml ammonium acetate solution or ammonium sulphate solution.

For exhaustion, add additional amount of 1 ml acetic acid solution.

- c) *1:1 metal complex acid dyes* — 5 ml sodium sulphate (calcined) solution and 4.0 ml sulphuric acid solution.
- d) *2:1 metal complex acid dyes* — 1.0 ml ammonium sulphate solution.

NOTE 2 — During dyeing, water should be added to make up the volume lost by evaporation.

A-4.2.2 Similarly, pipette out separately 45.0, 47.5, 50.0, 52.5 and 55.0 ml of solution of dyestuff under test in the dye vessels so as to give 0.90, 0.95, 1.00, 1.05 and 1.10 percent depths of shade (in terms of equivalent standard strength). Dye the hanks by following the method given in **A-4.2.1** (see Note).

NOTE — The dyeings with the solutions of the dyestuffs under test and of the standard dyestuff should be done simultaneously in the same water-bath.

A-4.3 Exhaust Dyeing — Make up the volumes of the exhaust liquor (see **A-4.2.1** and **A-4.2.2**) to the original volume (300 ml). Enter the fresh wetted hanks in the exhausted dye-baths and continue the dyeing for 30 minutes, at boil. Squeeze the hanks, rinse in warm water (50 to 60°C) and dry.

INDIAN STANDARDS

ON

METHODS OF TEST FOR DYES

IS :

1688-1960	Procedure for determination of fastness of dyestuffs
1962-1961	Method for determination of fastness of dyestuffs to metals in the dye-bath: chromium salts
1968-1961	Method for determination of fastness of dyestuffs to metals in the dye-bath: iron and copper
3859-1966	Method for determination of strength of water soluble azo dyes by reduction with titanium trichloride
4394-1967	Method for evaluating strength of homogeneous vat dyestuffs
4459-1967	Method for determination of strength of direct dyestuffs by dyeing test
4471-1967	Methods for determination of strength of naphthols (azoic coupling components) (gravimetric and volumetric methods)
4472 (Part I)-1967	Methods for identification of application classes of dyes on textile materials: Part I Cotton and other cellulosic fibres
4472 (Part II)-1968	Methods for identification of application classes of dyes on textile materials: Part II Wool, silk and other protein fibres
4472 (Part III)-1973	Methods for identification of application of classes of dyes on textile materials: Part III Man-made fibres
4946-1968	Method for evaluation of strength and shade of naphthol
5970-1970	Method for estimation of strength (vat content) of solubilized vat dyestuffs
7447-1974	Method for evaluating strength of reactive dyes (dichlorotriazinyl type) by dyeing test
7448-1974	Method for evaluating strength of reactive dyes (monochlorotriazinyl type) by dyeing test
7842-1975	Method for evaluating strength of reactive dyes (vinyl sulphone type) by dyeing test
7843-1975	Method for evaluation strength and shade of acid dyes by dyeing test
7844-1975	Method for evaluation strength and shade of chrome dyes by dyeing test
7845-1975	Method for evaluating strength of reactive dyes (trichloropyrimidyl type) by dyeing test

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